



# SHELLER-GLOBE

August 9, 1988

Ms. Audrey Bimby, Esq.  
Assistant Regional Counsel  
U.S. Environmental Protection Agency  
Region VII  
726 Minnesota Avenue  
Kansas City, Kansas 66101

Dear Ms. Bimby:

As per our August 2, 1988 letter, attached you will find our Proposed Work Plan for the agreed upon sediment sampling and analysis at our Keokuk Plant.


We would appreciate your timely review of this Work Plan since we have tentatively scheduled for the week of August 15 to conduct the sampling. We will provide you at least 72 hours advance notice of sampling in order that you may have an EPA representative present during the sampling.

We are flexible on the number and the location of the samples to be taken. Should you want to change the number and/or the location of the samples, this could be accomplished through a telephone conversation.

If you have any questions on this matter, please feel free to contact myself or Ms. Diana Dutton (214-969-2855).

Sincerely,

SHELLER GLOBE CORPORATION

  
Gregory D. Sautter,  
Manager of Environmental  
Activities

GDS/jsc  
Attachment

cc: Ms. D. Dutton  
Mr. A. Edgar  
Mr. L. King  
Mr. R. Ransford



R00110909  
RCRA RECORDS CENTER

AUG 10 1988

SHELLER GLOBE CORPORATON

KEOKUK PLANT

Sediment Sampling Plan

Sheller Globe has agreed to sample sediments from the areas on its Keokuk Plant property known as the "basin", the "ditch", and the "cooling pond." These samples will then be analyzed for Cadmium using the extraction procedure toxicity ("EP Tox") test method.

This plan has been designed to standardize the method of obtaining representative samples of sediment. It is also intended to standardize documentation of sampling procedures and the processing of samples for laboratory testing. Implementation of this plan will begin upon approval from the U.S. EPA Region VII.

Sample Location

Sheller Globe proposes to take a total of eight (8) sediment samples; two (2) in the basin, two (2) in the ditch, and four (4) in the cooling pond.

The location of these samples will be as follows (shown on Figure 1):

- A) Directly below discharge of sewer outlet into the basin.
- B) At the outlet of the basin.
- C) Half-way down the ditch between the basin and the inlet of the culvert to the cooling pond.
- D) In the ditch at the inlet of the culvert to the cooling pond.
- E) In the cooling pond at the outlet of the culvert (entrance to the pond).
- F) In the cooling pond at approximately 50 ft. downstream from the culvert.
- G) In the cooling pond at approximately 75 ft. downstream from the culvert.
- H) In the cooling pond at approximately 100 ft. downstream from the culvert.

### Sampling Procedure

Sediment sampling will be done using 24 inch long by 2 inch diameter stainless steel hand core tube sampler. The core sampler will be driven into the bottom sediment to a depth of 24 inches. The sample will then be withdrawn and the sample tube removed from the handle. The sample will be extruded from the sample tube into either a stainless steel bucket or directly into the sample container.

Sampling will be conducted by an outside laboratory personnel. The samples will be transported to and analyzed by the same laboratory within 24 hours of sampling. Chain of custody of these samples shall be maintained and recorded by the laboratory personnel.

### Sample Analysis

An extract of each sediment sample will be obtained using the Extraction Procedure (EP) Toxicity Test Method (Method 1310, copy attached).

Each extract will then be analyzed for Cadmium using Method 7130 (copy attached).

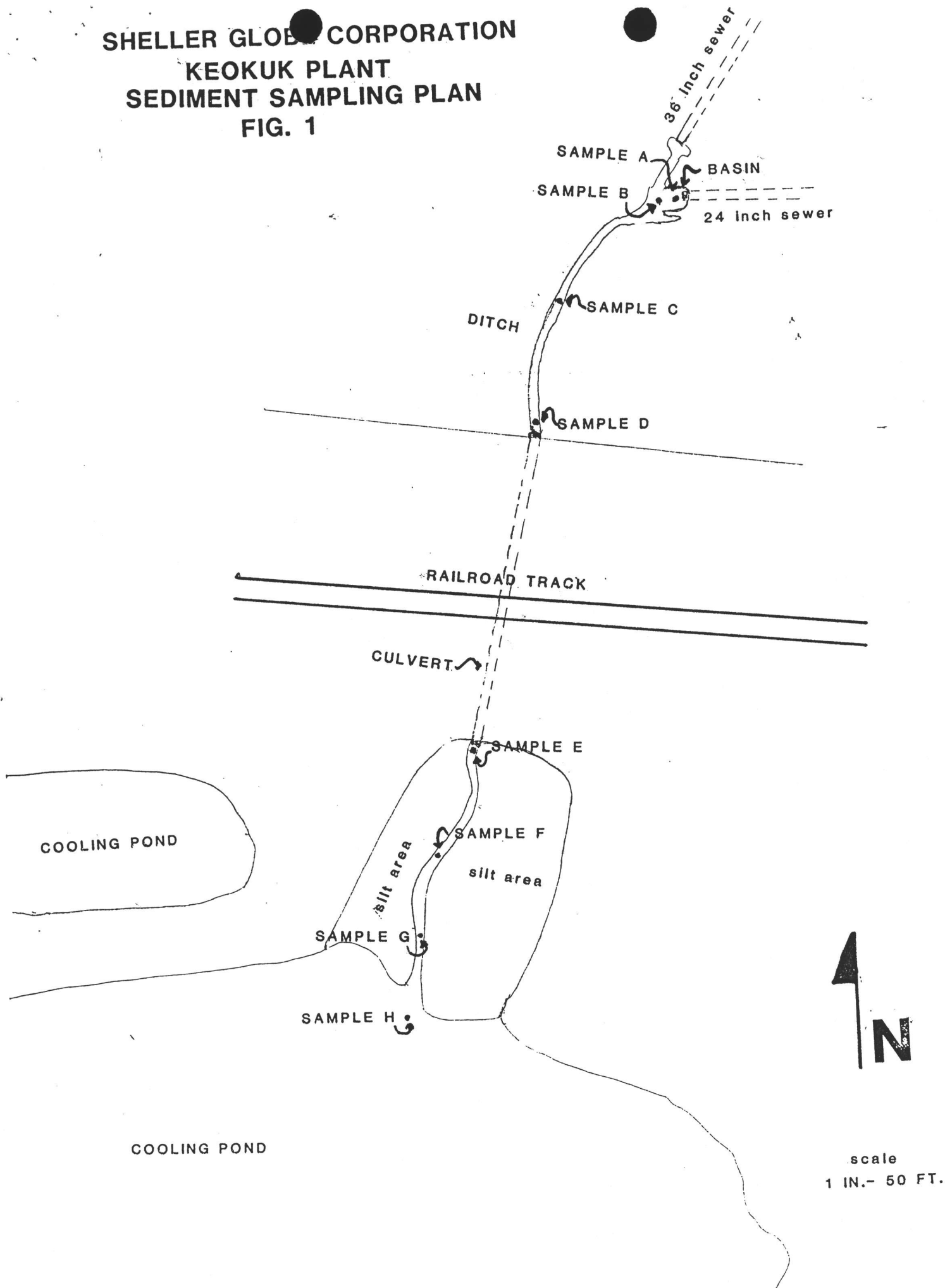
Documentation of all Quality Assurance (QA) and Quality Control (QC) of sampling, transporting and analyzing these samples shall be maintained and provided by the outside laboratory. Analytical results will be submitted to the U.S. EPA Region VII after QA/QC review.

### Decontamination

Sampling equipment will be completely decontaminated between samples to avoid cross-contamination between sampling locations. Equipment will be decontaminated near the sampling location, and set in plastic bags for transportation to the next sampling station. The decontamination method is as follows:

- 1) Wash tubing or sampler with hot water and a non-foaming detergent, such as trisodium phosphate.
- 2) Rinse with hot water.
- 3) Rinse with diluted 1:1 nitric acid.
- 4) Rinse with very hot water.
- 5) Rinse with deionized water.
- 6) Store the sampler in aluminum foil or plastic bag until ready for use.

SHELLER GLOBAL CORPORATION  
KEOKUK PLANT  
SEDIMENT SAMPLING PLAN  
FIG. 1



## METHOD 1310

### EXTRACTION PROCEDURE (EP) TOXICITY TEST METHOD AND STRUCTURAL INTEGRITY TEST

#### 1.0 Scope and Application

1.1 The extraction procedure (EP) described in this method is designed to simulate the leaching a waste will undergo if disposed of in an improperly designed sanitary landfill. Method 1310 is applicable to liquid, solid, and multiphasic samples.

#### 2.0 Summary of Method

2.1 If a representative sample of the waste contains more than 0.5% solids, the solid phase of the sample is extracted with deionized water which is maintained at a pH of  $5 \pm 0.2$  using acetic acid. The extract is analyzed to determine if any of the threshold limits listed in Table 1 are exceeded. Table 1 also specifies the approved method of analysis. Wastes that contain less than 0.5% solids are not subjected to extraction, but are directly analyzed and evaluated in a manner identical to that of extracts.

#### 3.0 Interferences

3.1 Potential interferences that may be encountered during analysis are discussed in the individual analytical methods referenced in Table 1.

#### 4.0 Apparatus and Materials

4.1 Extractor: For purposes of this test, an acceptable extractor is one that will impart sufficient agitation to the mixture to (1) prevent stratification of the sample and extraction fluid and (2) ensure that all sample surfaces are continuously brought into contact with well-mixed extraction fluid. Examples of suitable extractors are shown in Figures 1-3 of this method and Section 2.2 (Mobility) of this manual and are available from Associated Designs & Manufacturing Co., Alexandria, Virginia; Kraft Apparatus Inc., Mineola, New York; Millipore, Bedford, Massachusetts; and Rexnord, Milwaukee, Wisconsin.

4.2 pH Meter or pH Controller (Chemtrix, Inc., Hillsboro, Oregon is a possible source of a pH controller).

4.3 Filter holder: A filter holder capable of supporting a 0.45- $\mu$  filter membrane and able to withstand the pressure needed to accomplish separation. Suitable filter holders range from simple vacuum units to relatively complex systems that can exert up to 5.3 kg/cm<sup>2</sup> (75 psi) of pressure. The type of filter holder used depends upon the properties of the mixture to be filtered. Filter holders known to EPA and deemed suitable for use are listed in Table 2.

## 2 / CHARACTERISTICS - EP Toxicity

TABLE 1. MAXIMUM CONCENTRATION OF CONTAMINANTS  
FOR CHARACTERISTIC OF EP TOXICITY

Contaminant	Maximum concentration (mg/l)	Analytical method
Arsenic	5.0	7060, 7061
Barium	100.0	7080, 7081
Cadmium	1.0	7130, 7131
Chromium	5.0	7190, 7191 7195* 7196* 7197*
Lead	5.0	7420, 7421
Mercury	0.2	7470
Selenium	1.0	7740, 7741
Silver	5.0	7760, 7761
Endrin (1,2,3,4,10,10-Hexachloro-1 7-epoxy-1,4,4a,5,6,7,8,8a-octahydro-1 4-endo, endo-5,8-dimethanonaph- thalene)	0.02	8080
Lindane (1,2,3,4,5,6- Hexachlorocyclohexane, gamma isomer)	0.4	8080
Methoxychlor (1,1,1-Trichloro-2,2-bis (p-methoxyphenyl)ethane)	10.0	8080
Toxaphene (C <sub>10</sub> H <sub>10</sub> Cl <sub>8</sub> , Technical chlorinated camphene, 67-69% chlorine)	0.5	8080
2,4-D (2,4-Dichlorophenoxyacetic acid)	10.0	8150
2,4,5-TP (Silvex) (2,4,5- Trichlorophenoxypropionic acid)	1.0	8150

\* Methods used for determining hexavalent chromium

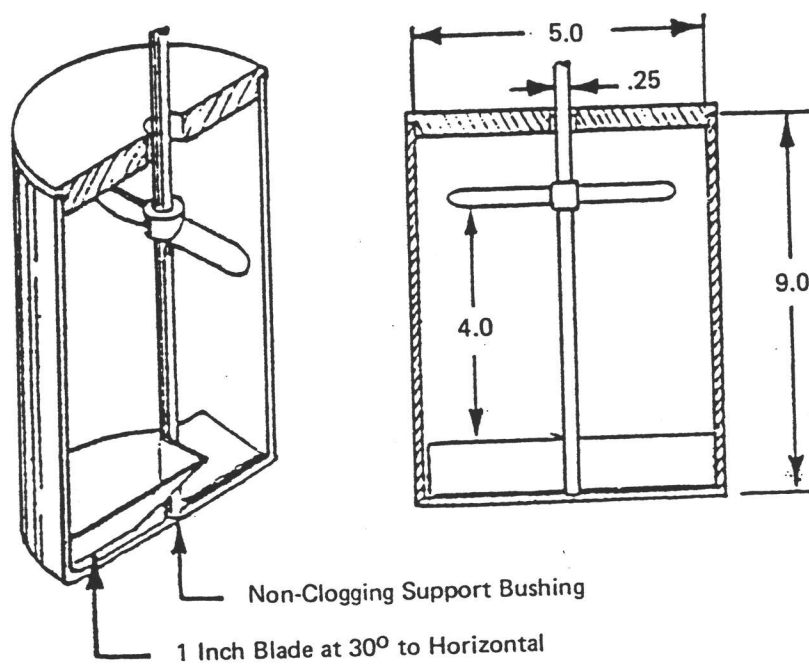


Figure 1. Extractor.

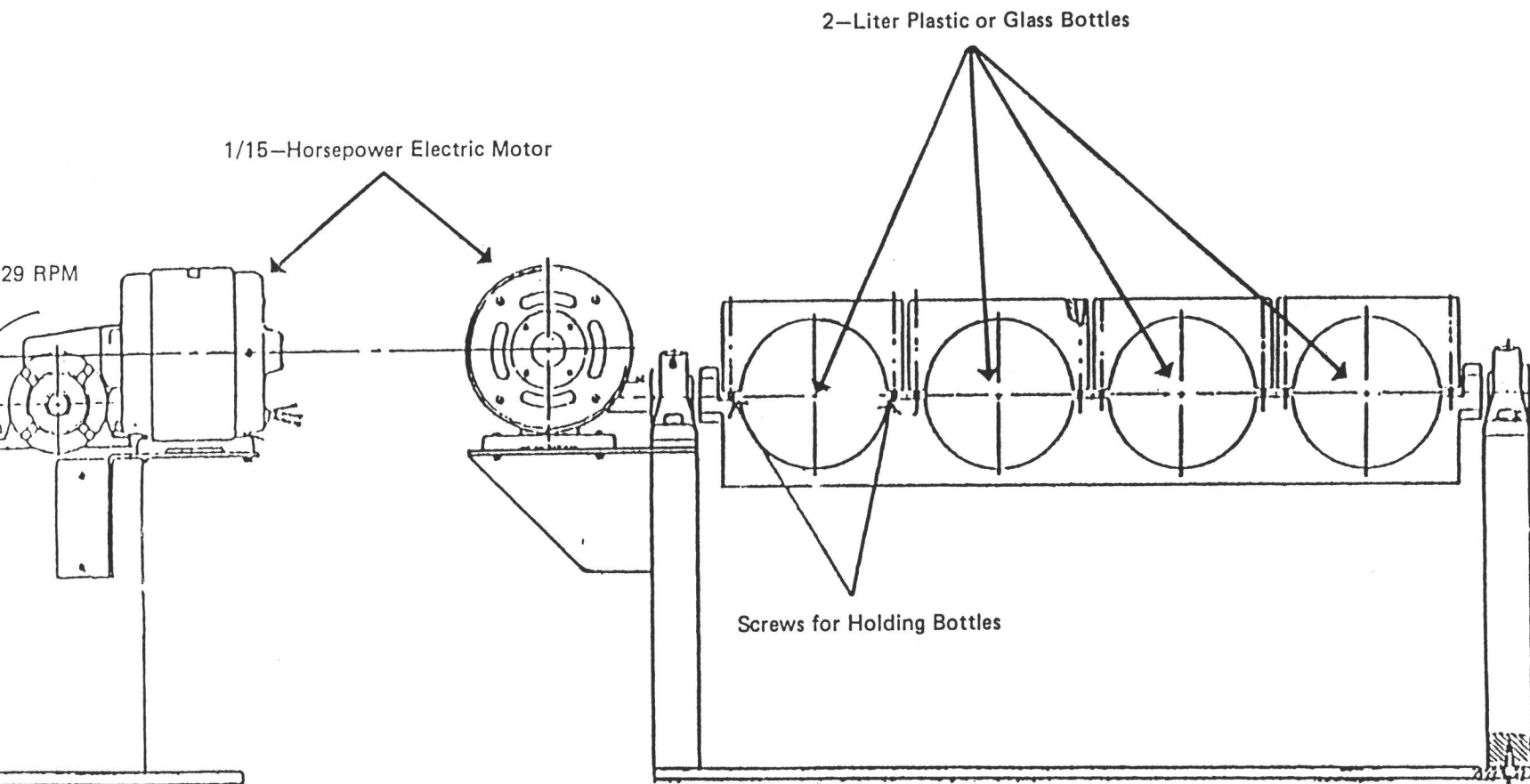


Figure 2. Rotary Extractor.



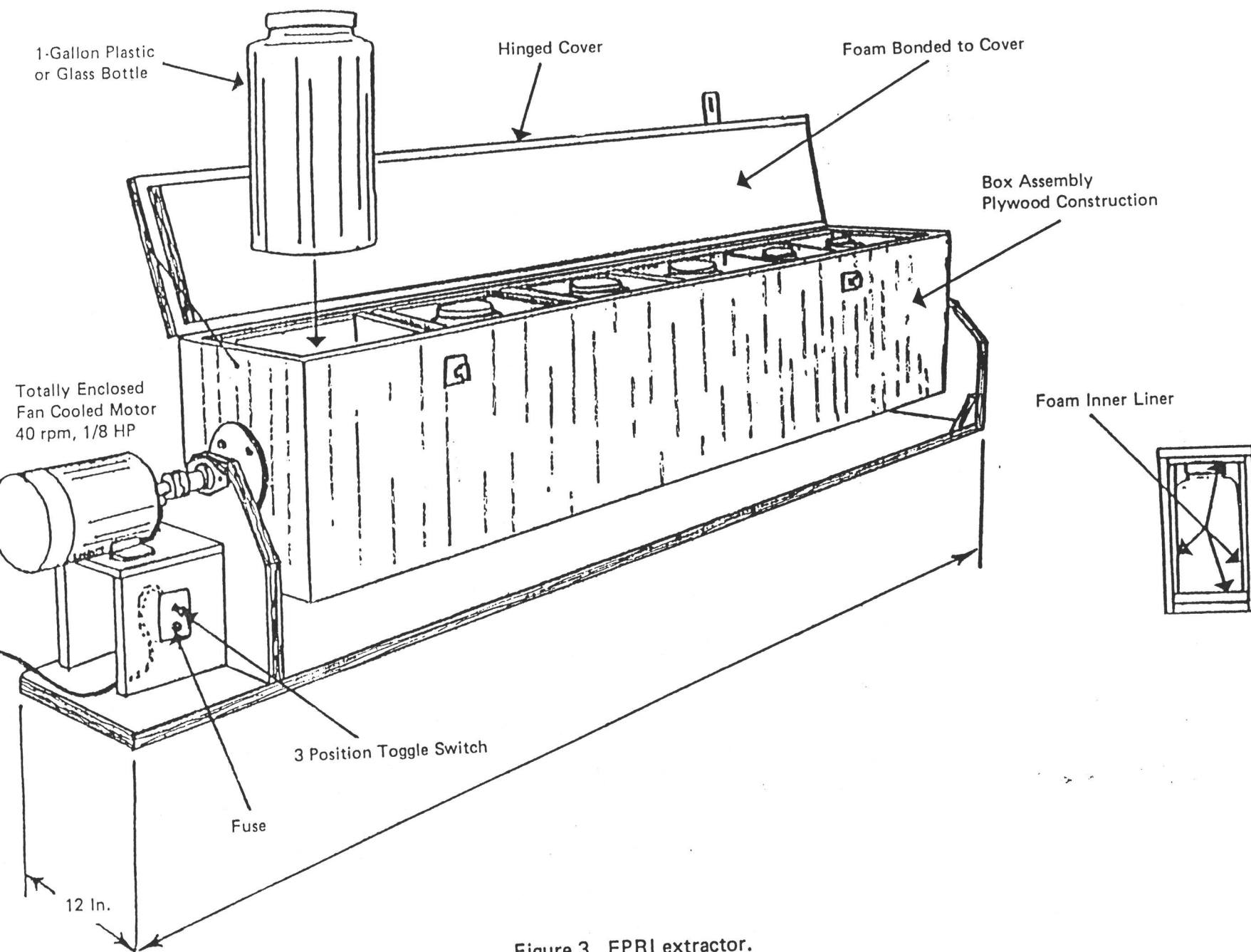


Figure 3. EPRI extractor.

## 6 / CHARACTERISTICS - EP Toxicity

4.4 Filter membrane: Filter membrane suitable for conducting the required filtration shall be fabricated from a material which: (1) is not physically changed by the waste material to be filtered, and (2) does not absorb or leach the chemical species for which a waste's EP Extract will be analyzed. Table 3 lists filter media known to the agency and generally found to be suitable for solid waste testing.

4.4.1 In cases of doubt, contact the filter manufacturer to determine if the membrane or the prefilter are adversely affected by the particular waste. If no information is available, submerge the filter in the waste's liquid phase. After 48 hr, a filter that undergoes visible physical change (i.e., curls, dissolves, shrinks, or swells) is unsuitable for use.

TABLE 2. EPA-APPROVED FILTER HOLDERS

Manufacturer	Size	Model No.	Comments
<u>Vacuum Filters</u>			
Nalgene	500 ml	44-0045	Disposable plastic unit, includes prefilter and filter pads, and reservoir; should be used when solution is to be analyzed for inorganic constituents
Nuclepore	47 mm	410400	
Millipore	47 mm	XX10 047 00	
<u>Pressure Filters</u>			
Nuclepore	142 mm	425900	
Micro Filtration Systems	142 mm	302300	
Millipore	142 mm	YT30 142 HW	

TABLE 3. EPA-APPROVED FILTRATION MEDIA

Supplier	Filter to be used for aqueous systems	Filter to be used for organic systems
<u>Coarse Prefilter</u>		
Gelman	61631, 61635	61631, 61635
Nuclepore	210907, 211707	210907, 211707
Millipore	AP25 035 00, AP25 127 50	AP25 035 00, AP25 127 50
<u>Medium prefilters</u>		
Nuclepore	210905, 211705	210905, 211705
Millipore	AP20 035 00, AP20 124 50	AP20 035 00, AP20 124 50
<u>Fine prefilters</u>		
Gelman	64798, 64803	64798, 64803
Nuclepore	210903, 211703	210903, 211703
Millipore	AP15 035 00, AP15 124 50	AP15 035 00, AP15 124 50
<u>Fine filters (0.45 <math>\mu</math>m)</u>		
Gelman	60173, 60177	60540 or 66149, 60544 or 66151
Pall	NX04750, NX14225	
Nuclepore	142218	142218 <sup>a</sup>
Millipore	HAWP 047 00, HAWP 142 50	FHUP 047 00, FHLP 142 50
Selas	83485-02, 83486-02	83485-02, 83486-02

<sup>a</sup>Susceptible to decomposition by certain polar organic solvents.

## 8 / CHARACTERISTICS - EP Toxicity

4.4.2.1 Prepare a standard solution of the chemical species of interest.

4.4.2.2 Analyze the standard for its concentration of the chemical species.

4.4.2.3 Filter the standard and re-analyze. If the concentration of the filtrate differs from the original standard, the filter membrane leaches or absorbs one or more of the chemical species.

4.5 Structural integrity tester: Having a 3.18-cm (1.25-in.) diameter hammer weighing 0.33 kg (0.73 lb) and having a free fall of 15.24 cm (6 in.) shall be used. This device is available from Associated Design and Manufacturing Company, Alexandria, VA 22314, as Part No. 125, or it may be fabricated to meet the specifications shown in Figure 4.

### 5.0 Reagents

5.1 Deionized water: Water should be monitored for impurities.

5.2 0.5 N acetic acid: This can be made by diluting concentrated glacial acetic acid (17.5 N). The glacial acetic acid should be of high purity and monitored for impurities.

5.3 Analytical standards should be prepared according to the analytical methods referenced in Table 1.

### 6.0 Sample Collection, Preservation and Handling

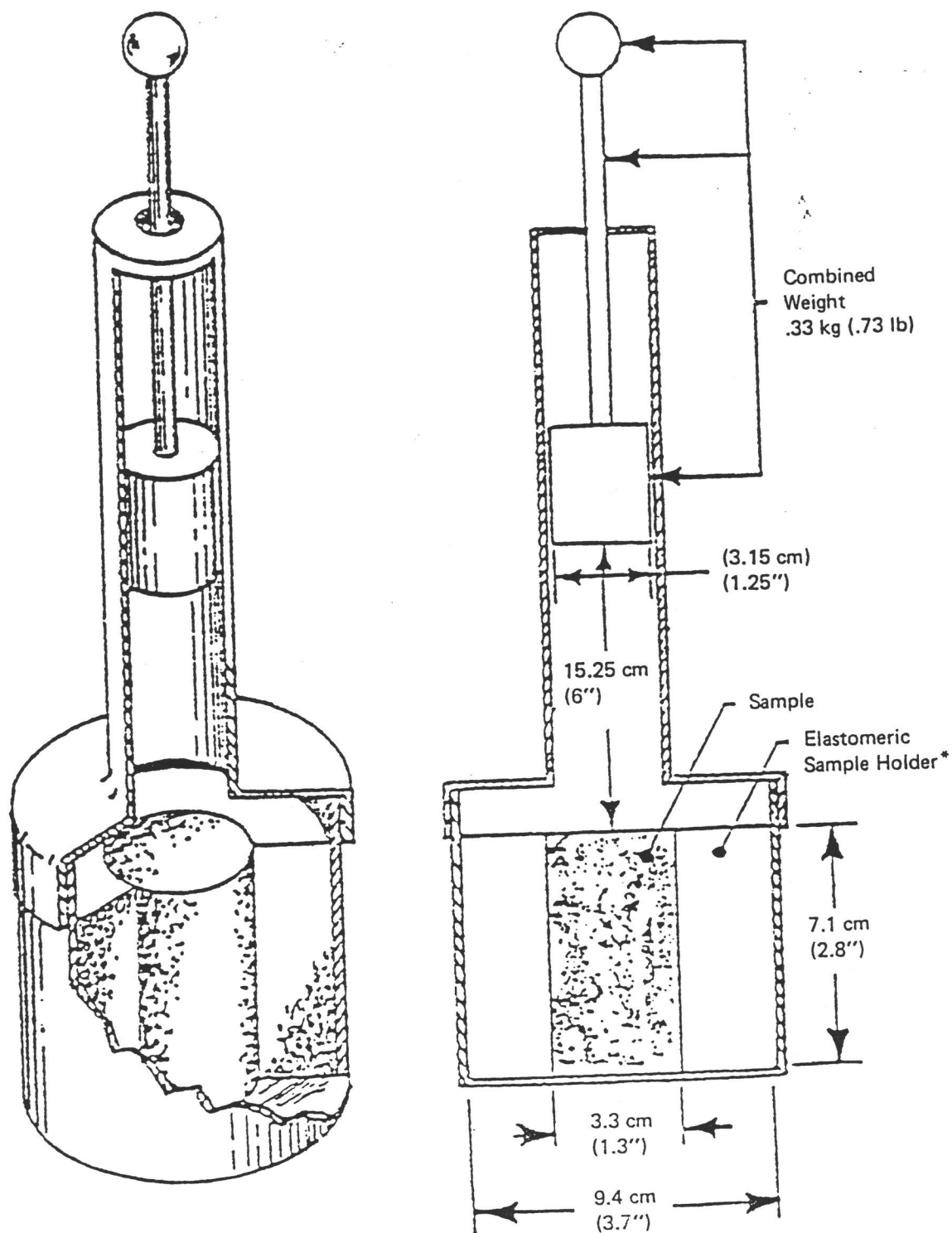
6.1 All samples must be collected using a sampling plan that addresses the considerations discussed in Section One of this manual.

6.2 Preservatives must not be added to samples.

6.3 Samples can be refrigerated if it is determined that refrigeration will not affect the integrity of the sample.

### 7.0 Procedure

7.1 If the waste does not contain any free liquid, go to Section 7.9. If the sample is liquid or multiphase, continue as follows. Weigh filter membrane and prefilter to  $\pm 0.01$  g. Handle membrane and prefilters with blunt curved-tip forceps or vacuum tweezers, or by applying suction with a pipette.



\* Elastomeric sample holder fabricated of material firm enough to support the sample.

Figure 4. Compaction tester.

## 10 / CHARACTERISTICS - EP Toxicity

7.2 Assemble filter holder, membranes, and prefilters following the manufacturer's instructions. Place the 0.45- $\mu$ m membrane on the support screen and add prefilters in ascending order of pore size. Do not prewet filter membrane.

7.3 Weigh out a representative subsample of the waste (100 g minimum).

7.4 Allow slurries to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged prior to filtration.

7.5 Wet the filter with a small portion of the waste's or extraction mixture's liquid phase. Transfer the remaining material to the filter holder and apply vacuum or gentle pressure (10-15 psi) until all liquid passes through the filter. Stop filtration when air or pressurizing gas moves through the membrane. If this point is not reached under vacuum or gentle pressure, slowly increase the pressure in 10-psi increments to 75 psi. Halt filtration when liquid flow stops. This liquid will constitute part or all of the extract (refer to Section 7.16). The liquid should be refrigerated until time of analysis.

NOTE: Oil samples or samples which contain oil are treated in exactly the same way as any other sample. The liquid portion of the sample is filtered and treated as part of the EP extract. If the liquid portion of the sample will not filter (this is usually the case with heavy oils or greases) it is carried through the EP extraction as a solid.

7.6 Remove the solid phase and filter media and, while not allowing it to dry, weight to  $\pm 0.01$  g. The wet weight of the residue is determined by calculating the weight difference between the weight of the filters (Section 7.1) and the weight of the solid phase and the filter media.

7.7 The waste will be handled differently from this point on depending on whether it contains more or less than 0.5% solids. If the sample appears to have less than 0.5% solids, the percent solids will be determined by the following procedure.

7.7.1 Dry the filter and residue at 80° C until two successive weighings yield the same value.

7.7.2 Calculate the percent solids using the following equation:

$$\frac{\text{weight of filtered solid and filters} - \text{tared weight of filters}}{\text{initial weight of waste material}} \times 100 = \% \text{ solids}$$

NOTE: This procedure is only used to determine whether the solid must be extracted or whether it can be discarded unextracted. It

is not used in calculating the amount of water or acid to use in the extraction step. Do not extract solid material that has been dried at 80° C. A new sample will have to be used for extraction if a percent solids determination is performed.

7.8 If the solid comprises less than 0.5% of the waste, discard the solid and proceed immediately to Section 7.17, treating the liquid phase as the extract.

7.9 The solid material obtained from Section 7.5 and all materials that do not contain free liquids should be evaluated for particle size. If the solid material has a surface area per gram of material equal to or greater than 3.1 cm<sup>2</sup> or passes through a 9.5-mm (0.375-in.) standard sieve, the operator should proceed to Section 7.11. If the surface area is smaller or the particle size larger than specified above, the solid material would be prepared for extraction by crushing, cutting or grinding the material so that it passes through a 9.5-mm (0.375-in.) sieve or, if the material is in a single piece, by subjecting the material to the "Structural Integrity Procedure" described in Section 7.10.

#### 7.10 Structural Integrity Procedure (SIP):

7.10.1 Cut a 3.3-cm-diameter by 7.1-cm-long cylinder from the waste material. For wastes that have been treated using a fixation process, the waste may be cast in the form of a cylinder and allowed to cure for 30 days prior to testing.

7.10.2 Place waste into sample holder and assemble the tester. Raise the hammer to its maximum height and drop. Repeat 14 additional times.

7.10.3 Remove solid material from tester and scrape off any particles adhering to sample holder. Weigh the waste to the nearest 0.01 g and transfer it to the Extractor.

7.11 If the sample contains more than 0.5% solids, use the wet weight of the solid phase obtained in Section 7.6 for purposes of calculating the amount of liquid and acid to employ for extraction by using the following equation:

$$W = W_f - W_t$$

where:

W = wet weight in grams of solid to be charged to extractor

W<sub>f</sub> = wet weight in grams of filtered solids and filter media

W<sub>t</sub> = weight in grams of tared filters.

## 12 / CHARACTERISTICS - EP Toxicity

If the waste does not contain any free liquids, 100 g of the material will be subjected to the extraction procedure.

7.12 Place the appropriate amount of material (refer to Section 7.11) into the extractor and add 16 times its weight of deionized water.

7.13 After the solid material and deionized water are placed in the extractor, the operator should begin agitation and measure the pH of the solution in the extractor. If the pH is greater than 5.0, the pH of the solution should be decreased to  $5.0 \pm 0.2$  by adding 0.5 N acetic acid. If the pH is equal to or less than 5.0, no acetic acid should be added. The pH of the solution should be monitored, as described below, during the course of the extraction and, if the pH rises above 5.2, 0.5 N acetic acid should be added to bring the pH down to  $5.0 \pm 0.2$ . However, in no event shall the aggregate amount of acid added to the solution exceed 4 ml of acid per gram of solid. The mixture should be agitated for 24 hr and maintained at  $20^{\circ}\text{--}40^{\circ}\text{ C}$  ( $68^{\circ}\text{--}104^{\circ}\text{ F}$ ) during this time. It is recommended that the operator monitor and adjust the pH during the course of the extraction with a device such as the Type 45-A pH Controller manufactured by Chemtrix, Inc., Hillsboro, Oregon 97123 or its equivalent, in conjunction with a metering pump and reservoir of 0.5 N acetic acid. If such a system is not available, the following manual procedure shall be employed.

7.13.1 A pH meter should be calibrated in accordance with the manufacturer's specifications.

7.13.2 The pH of the solution should be checked and, if necessary, 0.5 N acetic acid should be manually added to the extractor until the pH reaches  $5.0 \pm 0.2$ . The pH of the solution should be adjusted at 15-, 30-, and 60-min intervals, moving to the next longer interval if the pH does not have to be adjusted more than 0.5 pH units.

7.13.3 The adjustment procedure should be continued for at least 6 hr.

7.13.4 If, at the end of the 24-hr extraction period, the pH of the solution is not below 5.2 and the maximum amount of acid (4 ml per gram of solids) has not been added, the pH should be adjusted to  $5.0 \pm 0.2$  and the extraction continued for an additional 4 hr, during which the pH should be adjusted at 1-hr intervals.

7.14 At the end of the extraction period, deionized water should be added to the extractor in an amount determined by the following equation:

$$V = (20)(W) - 16(W) - A$$

where:

V = ml deionized water to be added

W = weight in g of solid charged to extractor

A = ml of 0.5 N acetic acid added during extraction



7.15 The material in the extractor should be separated into its component liquid and solid phases in the following manner.

7.15.1 Allow slurries to stand to permit the solid phase to settle (wastes that are slow to settle may be centrifuged prior to filtration) and set up the filter apparatus (refer to Section 4.3 and 4.4).

7.15.2 Wet the filter with a small portion of the waste's or extraction mixture's liquid phase. Transfer the remaining material to the filter holder and apply vacuum or gentle pressure (10-15 psi) until all liquid passes through the filter. Stop filtration when air or pressurizing gas moves through the membrane. If this point is not reached under vacuum or gentle pressure, slowly increase the pressure in 10 psi increments to 75 psi. Halt filtration when liquid flow stops.

7.16 The liquids resulting from Sections 7.5 and 7.15 should be combined. This combined liquid (or the waste itself if it has less than 0.5% solids, as noted in Section 7.8) is the extract and should be analyzed for the presence of any of the contaminants specified in Table 1 using the Analytical Procedures designated in Section 7.17.

7.17 The extract will be prepared and analyzed according to the analytical methods specified in Table 1. All of these analytical methods are included in this manual. The method of standard addition will be employed for all metal analyses.

NOTE: If the EP extract includes two phases, concentration of contaminants is determined by using a simple weighted average. For example: An EP extract contains 50 ml of oil and 1,000 ml of an aqueous phase. Contaminant concentrations are determined for each phase. The final contamination concentration is taken to be

$$\frac{(50)(\text{contaminant conc. in oil}) + (1,000)(\text{contaminant conc. of aqueous phase})}{1,050}$$

7.18 The extract concentrations are compared to the maximum contamination limits listed in Table 1. If the extract concentrations are equal to or greater than the respective values, then the waste is considered to be EP toxic.<sup>1</sup>

<sup>1</sup>Chromium concentrations have to be interpreted differently. A waste containing chromium will be determined to be EP toxic if (1) the waste extract has an initial pH of less than 7 and contains more than 5 mg/l of hexavalent chromium in the resulting extract, or (2) the waste extract has an initial pH greater than 7 and a final pH greater than 7 and contains more than 5 mg/l of hexavalent chromium in the extract, or (3) the waste extract has an initial pH greater than 7 and a final pH less than 7 and contains more than 5 mg/l of total chromium, unless the chromium is trivalent. To determine whether the chromium is trivalent, the sample must be processed according to an alkaline digestion method (Method 3060) and analyzed for hexavalent chromium (Methods 7195, 7196, or 7197).

14 / CHARACTERISTICS - EP Toxicity; MOBILITY

8.0 Quality Control

8.1 All quality control data should be maintained and available for easy reference or inspection.

8.2 Employ a minimum of one blank per sample batch to determine if contamination or any memory effects are occurring.

8.3 All quality control measures suggested in the referenced analytical methods should be followed.

## METHOD 7130

### CADMIUM (ATOMIC ABSORPTION, DIRECT ASPIRATION METHOD)

#### 1.0 Scope and Application

1.1 Method 7130 is an atomic absorption procedure approved for determining the concentration of cadmium in wastes, mobility procedure extracts, soils, and groundwater. All samples must be subjected to an appropriate dissolution step prior to analysis.

#### 2.0 Summary of Method

2.1 Prior to analysis by Method 7130, samples must be prepared for direct aspiration. The method of sample preparation will vary according to the sample matrix. Aqueous samples are subjected to acid digestion procedure (Method 3010). Sludge samples are prepared using the procedure described in Method 3050. For samples containing oils, greases, or waxes, the procedures described in Methods 3030 and 3040 may be applicable.

2.2 Following the appropriate dissolution of the sample, a representative aliquot is aspirated into an air/acetylene flame. The resulting absorption of hollow cathode radiation will be proportional to the cadmium concentration. Background correction must be employed for all analyses.

2.3 The typical detection limit for this method is 0.005 mg/l; typical sensitivity is 0.025 mg/l.

#### 3.0 Interferences

3.1 Nonspecific absorption and light scattering can be significant at the analytical wavelength. Thus background correction is required.

3.2 Samples and standards should be monitored for viscosity differences that may alter the aspiration rate.

#### 4.0 Apparatus and Materials

4.1 Atomic absorption spectrophotometer: Single or dual channel, single- or double-beam instrument, having a grating monochromator, photomultiplier detector, adjustable slits, and provisions for background correction.

4.2 Cadmium hollow cathode lamp or electrodeless discharge lamp.

4.3 Strip chart recorder (optional).

## 2 / INORGANIC ANALYTICAL METHODS

### 5.0 Reagents

5.1 ASTM Type II water (ASTM D1193): Water should be monitored for impurities.

5.2 Concentrated nitric acid: Acid should be analyzed to determine level of impurities. If impurities are detected, all analyses should be blank-corrected.

5.3 Cadmium standard stock solution (1000 mg/l): Either procure a certified aqueous standard from a supplier (Spex Industries, Alpha Products, or Fisher Scientific) and verify by comparison with a second standard, or dissolve 2.282 g cadmium sulfate ( $\text{CdSO}_4 \cdot 8\text{H}_2\text{O}$ , analytical reagent grade) and dissolve in Type II water or equivalent.

5.4 Cadmium working standards: These standards should be prepared with the same type and same concentration of acid that will be found in the analytical solution.

5.5 Air: Cleaned and dried through a suitable filter to remove oil, water, and other foreign substances. The source may be a compressor or a cylinder of industrial-grade compressed air.

5.6 Acetylene: Should be of high purity. Acetone, which is usually present in acetylene cylinders, can be prevented from entering and affecting flame conditions, by replacing the cylinder before the pressure has fallen to 50 psig.

### 6.0 Sample Collection, Preservation, and Handling

6.1 All samples must have been collected using a sampling plan that addresses the considerations discussed in Section One of this manual.

6.2 All sample containers must be prewashed with detergents, acids, and Type II water. Plastic and glass containers are both suitable.

6.3 Aqueous samples must be acidified to a pH of less than 2 with nitric acid.

6.4 Nonaqueous samples shall be refrigerated when possible, and analyzed as soon as possible.

### 7.0 Procedure

7.1 Sample preparation: Aqueous samples should be prepared according to Method 3010; sludge-type samples should be prepared according to Method 3050; and samples containing oils, greases or waxes may be prepared according

to Methods 3030 or 3040. The applicability of a sample preparation technique to a new matrix type must be demonstrated by analyzing spiked samples and/or relevant standard reference materials.

7.2 The 228.8-nm wavelength line and background correction shall be employed.

7.3 An oxidizing air/acetylene flame shall be used.

7.4 Follow the manufacturer's operating instructions for all other instrument parameters.

7.5 Either (1) run a series of cadmium standards and construct a calibration curve by plotting the concentrations of the standards against the absorbances or (2) for the method of standard additions, plot added concentration versus absorbance. For instruments that read directly in concentration, set the curve corrector to read out the proper concentration.

7.6 Analyze, by the method of standard additions, all EP extracts, all samples analyzed as part of a delisting petition, and all samples that suffer from matrix interferences.

7.7 Duplicates, spiked samples, and check standards should be routinely analyzed.

7.8 Calculate metal concentrations by (1) the method of standard additions, or (2) from a calibration curve, or (3) directly from the instrument's concentration readout. All dilution or concentration factors must be taken into account. Concentrations reported for multiphased or wet samples must be appropriately qualified (e.g., 5 µg/g dry weight).

## 8.0 Quality Control

8.1 All quality control data should be maintained and available for easy reference or inspection.

8.2 Calibration curves must be composed of a minimum of a blank and three standards. A calibration curve should be made for every hour of continuous sample analysis.

8.3 Dilute samples if they are more concentrated than the highest standard or if they fall on the plateau of a calibration curve.

8.4 Employ a minimum of one blank per sample batch to determine if contamination or any memory effects are occurring.

8.5 Analyze check standards after approximately every 15 samples.

#### 4 / INORGANIC ANALYTICAL METHODS

8.6 Run one duplicate sample for every 10 samples. A duplicate sample is a sample brought through the whole sample preparation process.

8.7 Spiked samples or standard reference materials shall be periodically employed to ensure that correct procedures are being followed and that all equipment is operating properly.

8.8 The method of standard additions shall be used for the analysis of all EP extracts, on all analyses submitted as part of a delisting petition, and whenever a new sample matrix is being analyzed.